04-04-01

(TO-1390 -93) U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C.371

ATTORNEY'S DOCKET NUMBER	
P1998J096	
U.S APPLICATION NO. (If known, see 37 CFR 1.5)	09/806873

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/US98/21595	13 October 1998	None

TITLE OF INVENTION

LONG LIFE GAS ENGINE OIL AND ADDITIVE SYSTEM

APPLICANT(S) FOR DO/EO/US

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Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. [X] This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. [ ] This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. [ ] This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(l).
4. [X] A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.

5. [X] A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   a. [X] is transmitted herewith (required only if not transmitted by the International Bureau)
   b. [ ] has been transmitted by the International Bureau
   c. [ ] is not required, as the application was filed in the United States Receiving Office (RO/US)
6. [ ] A translation of the International Application into English (35 U.S.C. 371(c)(2)).

7. [ ] Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. [ ] are transmitted herewtih (required only if not transmitted by the International Bureau).
   b. [ ] have been transmitted by the International Bureau.
   c. [ ] have not been made; however, the time limit for making such amendments has NOT expired.
   d. [ ] have not been made and will not be made.
8. [ ] A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).

9. [X] An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).

10. [ ] A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. [ ] An Information Disclosure Statement under 37 CVFR 1.97 and 1.98.

12. [ ] An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.

13. [ ] A FIRST preliminary amendment.
    [ ] A SECOND or SUBSEQUENT preliminary amendment.

14. [ ] A substitute specification.

15. [ ] A change of power of attorney and/or address letter.

16. [X] Other items or information:
    - X PCT Request form
    - X Notification of the International Application Number and of the International Filing Date
    - X Invitation to Correct Defect and Response
    - X Amendments Made During Chpter 1 (Art. 19) 6 sheets
    - X Notification of the Recording of a Change Form PCT/IB/306
    - X International Search Report (PCT/ISA/220)
    - X PCT Application as Published
    - X Notice Informing the Applicant of the Communication of the International Application to the Designated Offices
    - X Written Opinion Form PCT/IPEA/408 and No Response
    - X International Preliminary Examination Report (PCT/IPEA/416)
    - X References in Search Report US Patent 5,744,430; US Patent 3,396,114; US Patent 5,108,634 and US Patent 4,812,246

(July 1993)

U.S. APPLICATION NO. (if known, see 37 CFR 1.5)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER
09/806873	PCT/US98/21595	P1998J096

17. The following fees are submitted:

	CALCULATIONS	PTO USE ONLY
Basic National Fee (37 CFR 1.492(a)(1)-(5)):		
Search Report has been prepared by the EPO or JPO $846.00		
International preliminary examination fee paid to USPTO (37 CFR 1.482) $690.00		
No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) $700.00		
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO $1000.00		
International preliminary examination fee paid to USPTO (37 CFR .482) and all claims satisfied provisions of PCT Article 33(2)-(4)..... $94.00		
ENTER APPROPRIATE BASIC FEE AMOUNT =	$ 1000.00	
Surcharge of **$130.00** for furnishing the oath or declaration later than [ ] 20 [X] 30 Months from the earliest claimed priority date (37 CFR 1.492(e)).	$	

Claims	Number Filed		Number Extra	Rate		
Total Claims	16	- 20 =	0	x $18.00	$	0
Independent Claims	2	- 3 =	0	x $80.00	$	0
Multiple dependent claim(s) (if applicable)				+$270.00	$	270.00

TOTAL OF ABOVE CALCULATIONS =	$	1,270.00
Reduction by 1/2 for filing by small entity, if applicable. Verified Small Entity statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28).	$	-
SUBTOTAL =	$	1,270.00
Processing fee of **$130.00** for furnishing the English translation later than [ ] 20 [ ] 30 months from the earliest claimed priority date (37 CFR 1.492(f)).	$	
TOTAL NATIONAL FEE =	$	1,270.00
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). **$40.00** per property +	$	
TOTAL FEE ENCLOSED =	$	1,270.00
Amount to be: Refunded	$	
Charged	$	

a. [ ] A check in the amount of $ _____ to cover the above fees is enclosed.

b. [X] Please charge my Deposit Account No. 05-1330 in the amount of $ 1,270.00 To cover the above fees. A duplicate copy of this sheet is enclosed.

c. [X] The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 05-1330. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

EXXONMOBIL RESEARCH AND ENGINEERING COMPANY
P.O. Box 900
ANNANDALE, NEW JERSEY 08801-0900

Joseph J. Allocca
SIGNATURE

Joseph J. Allocca
NAME

☐ Pursuant to 37 CFR 1.34(a)

REGISTRATION NUMBER: 27,766

Form PTO-1390 (REV 5-93)

PCT INTERNATIONAL APPLICATION TRANSMITTAL LETTER	DATE April 3, 2001
REGARDING THE INTERNATIONAL APPLICATION OF EXXON RESEARCH AND ENGINEERING COMPANY	DOCKET OR REFERENCE NUMBER P1998J096
ENTITLED LONG LIFE GAS ENGINE OIL AND ADDITIVE SYSTEM	

Certification under 37 CFR 1.10 (if applicable)

EJ083186053US
"Express Mail" mailing number

April 3, 2001
Date of Deposit

I hereby certify that this application is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to the Assistant Commissioner for Patents, "Box PCT" , Washington, D.C. 20231.

Teresa L. Lachowski
(Typed or printed name of person mailing application)

Teresa L. Lachowski
(Signature of person mailing application)

To the United States Receiving Office (RO/US):
Accompanying this transmittal letter is the above-identified International application, including a completed Request form (PCT/RO/101). Please process the application according to the provisions of the Patent Cooperation Treaty.

LONG LIFE GAS ENGINE OIL AND ADDITIVE SYSTEM

FIELD OF THE INVENTION

The present invention relates to gas engine oils of enhanced life as evidenced by a reduction in viscosity increase, oxidation, nitration, TAN increase, and TBN depletion comprising a base oil of lubricating viscosity and a particular combination of anti oxidants and viscosity index improver additives.

BACKGROUND OF THE INVENTION

Natural gas fired engines are large, having up to 16 cylinders, and often generating between 500-3000 HP. The engines are typically used in the Oil and Gas industry to compress natural gas at well heads and along pipelines. Due to the nature of this application, the engines often run continuously near full load conditions, shutting down only for maintenance such as for oil changes. This condition of running continuously near full load places severe demands on the lubricant. Indeed, since the lubricant is subjected to a high temperature environment, the life of the lubricant is often limited by oil oxidation processes. Additionally, since natural gas fired engines run with high emissions of oxides of nitrogen (NO_X), the lubricant life may also be limited by oil nitration processes. Therefore, it is desirable for gas engine oils to have long life through enhanced resistance to oil oxidation and nitration.

The combustion of diesel fuel often results in a small amount of incomplete combustion (e.g., exhaust particulates). The incombustibles provide a small but critical degree of lubrication to the exhaust valve/seat interface, thereby ensuring the durability of both cylinder heads and valves. The combustion of natural gas is often very complete, with virtually no incombustible

materials. Therefore, the durability of the cylinder head and valve is controlled by the properties of the lubricant and its consumption rate. For this reason, gas engine oils are classified according to their ash content, since it is the lubricant ash which acts as a solid lubricant to protect the valve/seat interface. The oil industry has accepted guidelines which classify gas engine oils according to their ash level. The classifications are:

Ash Designation	Ash Level (wt%, ASTM D874)
Ashless	Ash < 0.1%
Low Ash	0.1 < Ash < 0.6
Medium Ash	0.6 < Ash < 1.5
High Ash	Ash > 1.5

The ash level of the lubricant is often determined by its formulation components, with metal-containing detergents (e.g., barium, calcium) and metallic-containing antiwear additives contributing to the ash level of the lubricant. For correct engine operation, gas engine manufacturers define lubricant ash requirements as part of the lubricant specifications. For example, manufacturers of 2-cycle engines often require the gas engine oil to be Ashless in order to minimize the extent of harmful deposits which form on the piston and combustion chamber area. Manufacturers of 4-cycle engines often require the gas engine oils to be Low, Medium or High Ash to provide the correct balance of engine cleanliness, and durability of the cylinder head and valves. Running the engine with too low an ash level will likely result in shortened life for the valves or cylinder head. Running the engine with too high an ash level will likely cause excessive deposits in the combustion chamber and upper piston area.

Gas engine oil of enhanced life as evidenced by an increase in the resistance of the oil to oxidation, nitration and deposit formation is the subject of

USP 5,726,133. The gas engine oil of that patent is a low ash gas engine oil comprising a major amount of a base oil of lubricating viscosity and a minor amount of an additive mixture comprising a mixture of detergents comprising at least one low Total Base Number (TBN) alkali or alkaline earth metal salt having a TBN of about 250 and less and a second alkali or alkaline earth metal salt having a TBN lower than the aforesaid component. The TBN of this second alkali or alkaline earth metal salt will typically be about half or less that of the aforesaid component.

The fully formulated gas engine oil of USP 5,726,133 can also typically contain other standard additives known to those skilled in the art, including dispersants (about 0.5 to 8 vol%), phenolic or aminic anti oxidants (about 0.05 to 1.5 vol%), metal deactivators such as triazoles, alkyl substituted dimercaptothiadiazoles (about 0.01 to 0.2 vol%), anti wear additives such as metal di thiophosphates, metal dithiocarbamates, metal xanthates or tricresyl-phosphates (about 0.05 to 1.5 vol%), pour point depressants such as poly (meth) acrylates or alkyl aromatic polymers (about 0.05-0.6 vol%), anti foamants such as silicone antifoaming agents (about 0.005 to 0.15 vol%), and viscosity index improvers, such as olefin copolymers, polymethacrylates, styrene-diene block copolmyers, and star copolymers (up to about 15 vol%, preferably up to about 10 vol%).

DESCRIPTION OF THE FIGURES

Figures 1A, 1B and 1C present engine tests comparing formulations of the present invention against prior art formulations in terms of viscosity increase, nitration levels and oxidation levels versus engine time (hours).

Figure 2 presents field test results in terms of drain interval (hours) for prior art commercial formulation (historic data) versus formulation of the present invention.

SUMMARY OF THE INVENTION

The present invention relates to a gas engine lubricating oil of enhanced life as evidenced by reductions in viscosity increase, oxidation, nitration, TAN increase, and TBN depletion, which comprises a major amount of a base oil of lubricating viscosity and a minor amount of a phenolic anti oxidant and a minor amount of viscosity index improver but which does not contain aminic anti oxidant.

DETAILED DESCRIPTION OF THE INVENTION

A gas engine oil is described comprising a major amount of a base oil of lubricating viscosity and a minor amount of phenolic type anti oxidant and viscosity index improver but which does not contain aminic anti oxidant. Also described is a method for enhancing the life of gas engine oils as evidenced by a reduction in viscosity increase, oxidation, nitration, TAN increase, and TBN depletion, by adding to a gas engine oil a minor amount of a phenolic anti oxidant and a viscosity index improver, but does not contain aminic anti oxidants.

The lubricating oil base stock is any natural or synthetic lubricating base oil stock fraction having a kinematic viscosity at 100°C of about 5 to 20 cSt, more preferably about 7 to 16 cSt, most preferably about 9 to 13 cSt. In a preferred embodiment the use of the viscosity index improver permits the omission of oil of vis about 20 cSt or more at 100°C from the lube base oil

fraction used to make the present formulation. Therefore, a preferred base oil is one which contains little, if any, heavy fraction, e.g., little, if any, lube oil fraction of viscosity 20 cSt or higher at 100°C.

The lubricating oil basestock can be derived from natural lubricating oils, synthetic lubricating oils or mixtures thereof. Suitable lubricating oil basestocks include basestocks obtained by isomerization of synthetic wax and slack wax, as well as hydrocrackate basestocks produced by hydrocracking (rather than solvent extracting) the aromatic and polar components of the crude.

Natural lubricating oils include animal oils, vegetable oils (e.g., rapeseed oils, castor oils and lard oil), petroleum oils, mineral oils, and oils derived from coal or shale.

Synthetic oils include hydrocarbon oils and halo-substituted hydrocarbon oils such as polymerized and inter-polymerized olefins, alkylbenzenes, polyphenyls, alkylated diphenyl ethers, alkylated diphenyl sulfides, as well as their derivatives, analogs and homologs thereof, and the like. Synthetic lubricating oils also include alkylene oxide polymers, interpolymers, copolymers and derivatives thereof wherein the terminal hydroxyl groups have been modified by esterification, etherification, etc. Another suitable class of synthetic lubricating oils comprises the esters of dicarboxylic acids with variety of alcohols. Esters useful as synthetic oils also include those made from C_5 to C_{12} monocarboxylic acids and polyols and polyol ethers.

Silicon-based oils (such as the polyakyl-, polyaryl-, polyalkoxy-, or polyaryloxy-siloxane oils and silicate oils) comprise another useful class of synthetic lubricating oils. Other synthetic lubricating oils include liquid esters of

phosphorus-containing acids, polymeric tetrahydrofurans, polyalphaolefins, and the like.

The lubricating oil may be derived from unrefined, refined, rerefined oils, or mixtures thereof. Unrefined oils are obtained directly from a natural source or synthetic source (e.g., coal, shale, or tar sand bitumen) without further purification or treatment. Examples of unrefined oils include a shale oil obtained directly from a retorting operation, a petroleum oil obtained directly from distillation, or an ester oil obtained directly from an esterification process, each of which is then used without further treatment. Refined oils are similar to the unrefined oils except that refined oils have been treated in one or more purification steps to improve one or more properties. Suitable purification techniques include distillation, hydrotreating, dewaxing, solvent extraction, acid or base extraction, filtration, and percolation, all of which are known to those skilled in the art. Rerefined oils are obtained by treating refined oils in processes similar to those used to obtain the refined oils. These rerefined oils are also known as reclaimed or reprocessed oils and often are additionally processed by techniques for removal of spent additives and oil breakdown products.

Lubricating oil base stocks derived from the hydroisomerization of wax may also be used, either alone or in combination with the aforesaid natural and/or synthetic base stocks. Such wax isomerate oil is produced by the hydro-isomerization of natural or synthetic waxes or mixtures thereof over a hydro-isomerization catalyst.

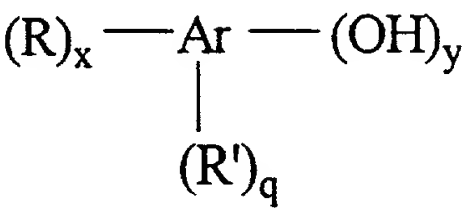
Natural waxes are typically the slack waxes recovered by the solvent dewaxing of mineral oils; synthetic waxes are typically the wax produced by the Fischer-Tropsch process.

The resulting isomerate product is typically subjected to solvent dewaxing and fractionation to recover various fractions of specific viscosity range. Wax isomerate is also characterized by possessing very high viscosity indices, generally having a VI of at least 130, preferably at least 135 and higher and following dewaxing, a pour point of about -20° C and lower.

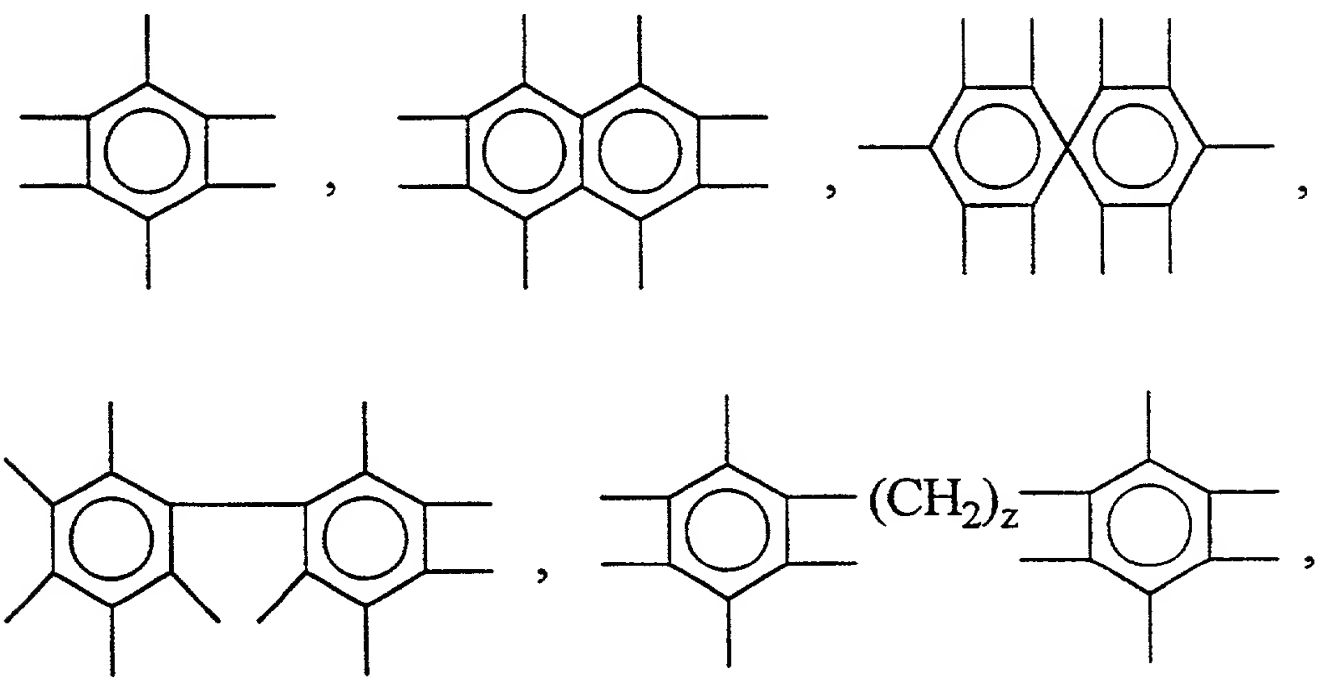
The production of wax isomerate oil meeting the requirements of the present invention is disclosed and claimed in U.S. Patent Nos. 4049,299 and 4,158,671.

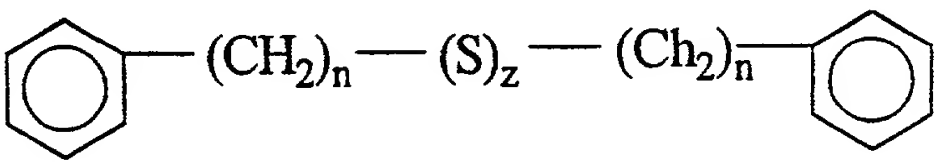
Phenolic anti oxidants useful in the present invention include any compounds containing hydroxy groups bound directly to an aromatic ring and which are substantially non-volatile at peak engine operating temperatures. By substantially non-volatile is meant that there is less than 10% phenol volatility at about 150°C, preferably at about 175°C, most preferably at about 200°C and higher. The term "phenol type" used herein includes compounds having one or more than one hydroxy group bound to an aromatic ring which may itself be mononuclear, e.g., benzyl, or polynuclear, e.g., naphthyl and spiro aromatic compounds. Thus "phenol type" includes phenol per se, catechol, resorcinol, hydroquinone, naphthol, etc., as well as alkyl or alkenyl and sulfurized alkyl or alkenyl derivatives thereof, and bisphenol type compounds including such bi-phenol compounds linked by alkylene bridges or sulfur or oxygen bridges. Alkyl phenol include mono- and poly-alkyl or alkenyl phenols, the alkyl or alkenyl group containing from about 3-100 carbons, preferably 4 to 50 carbons and sulfurized derivatives thereof, the number of alkyl or alkenyl groups present in the aromatic ring ranging from 1 to up to the available unsatisfied valences of the aromatic ring remaining after counting the number of hydroxyl groups bound to the aromatic ring.

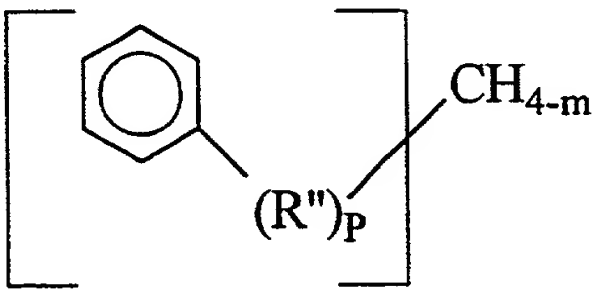
Generally, therefor, the "phenolic type" anti oxidant may be represented by the general formula:



where Ar is selected from the group consisting of:







wherein R is a C_3-C_{100} alkyl or alkenyl group, a sulfur substituted alkyl or alkenyl group, preferably a C_4-C_{50} alkyl or alkenyl group or sulfur substituted alkyl or alkenyl group, more preferably C_3-C_{100} alkyl or sulfur substituted

alkyl group, most preferably a C_4-C_{50} alkyl group, y ranges from 1 to up to the available valences of Ar, x ranges from 0 to up to the available valances of Ar-y, Q ranges from 0 to up to the available valences of Ar-(x + y + p), z ranges from 1 to 10, n ranges from 0 to 20, and m is 0 to 4 and P is 0 or 1, preferably y ranges from 1 to 3, x ranges from 0 to 3, z ranges from 1 to 4 and n ranges from 0 to 5, p is 0 and Q is 0 or 1.

Preferably, the phenolic type anti oxidant does not contain any N or S heteroatoms either as bridging moiety or as N or S containing substituted groups.

Most preferably the phenol is a hindered phenol such as di isopropyl phenol, di-tert butyl phenol, di tert butyl alkylated phenol where the alkyl substitutent is hydrocarbyl and contains between 1 and 20 carbon atoms, such as 2,6 di-tert butyl-4 methyl phenol, 2,6-di-tert butyl-4-ethyl phenol, etc., or 2,6 di-tert butyl 4-alkoxy phenol.

Phenolic type anti oxidants are well known in the lubricating industry and to those skilled in the art. The above is presented only by way of exemplification, not limitation on the type of phenolic anti oxidants which can be used in the present invention.

The amount of phenolic type anti oxidant used ranges from about 0.1 to 2 vol%, preferably about 0.3 to 1.75 V%, most preferably about 0.5 to 1.5 V%.

The oils of the present invention are essentially devoid of any aminic anti oxidants.

Viscosity index improvers useful in the present invention include any of the polymers which impact enhanced viscosity properties to the finished oil and are generally hydrocarbon-based polymers having a molecular weight, Mw, in the range of between about 2,000 to 1,000,000, preferably about 50,000 to 200,000. Viscosity index improver polymers typically include olefin copolymers, e.g., ethylene-propylene copolymers, ethylene-(iso-) butylene copolymers, propylene-(iso-) butylene copolymers, ethylene-poly alpha olefin copolymers, polymethocrylates; styrene-diene block copolymers, e.g., styrene-isoprene copolymers, and star copolymers. Viscosity index improvers may be monofunctional or multifuncational, such as those bearing substitutents that provide a secondary lubricant performance feature such as dispersancy, pour point depression, etc. In the present invention any multi functional viscosity index improver used will not possess aminic type anti oxidancy substitutents.

Viscosity index improvers are lubricant additives well known in the lubricant industry and to those skilled in the art. The above is presented only by way of example and not as a limitation on the types of viscosity index improvers which can be used in the present invention.

The amount of viscosity index improver used, be it mono functional or multifunctional, is in the amount of about 0.1 to 3 vol%, preferably about 0.2 to 2 V%, most preferably about 0.3 to 1.5 V%.

The fully formulated gas engine oil may contain additional, typical additives known to those skilled in the industry, used on an as-received basis.

Gas engine oils typically contain one or more detergents in varying amounts depending on whether the oil is classified as an ashless oil or a low, medium, or high ash content oil. The amount of detergent used and the type

used is left to the practitioner to decide based on the final gas engine oil ash specification target. Detergents typically include alkali or alkaline earth metal salts. These metal salts may have Total Base Numbers (TBN) between about 25 to 500, preferably between about 50 to 300. The metal salt will usually be metal phenates, sulfonates or salicylates. The metal will usually be sodium, magnesium or calcium, preferably calcium and magnesium.

A preferred low ash gas engine oil detergent system is disclosed and claimed in U.S. Patent 5,726,133 wherein the detergent comprises a mixture of detergents comprising at least one first alkali or alkaline earth metal salt or mixture thereof of low TBN of about 250 or less and at least one second alkali or alkaline earth metal salt or mixture thereof which is more neutral than the first low TBN salt. Preferably this second salt has a TBN of about one half or less that of the first salt.

The fully formulated oil may contain dispersants of the type generally represented by succinimides (e.g., polyisobutylene succinic acid/ anhydride (PIBSA)-polyamine having a PIBSA molecular weight of about 700 to 2500). The dispersants may be borated or non-borated. The dispersant can be present in the amount of about 0.5 to 8 vol%, more preferably in the amount of about 1 to 6 vol%, most preferably in the amount of about 2 to 4 vol%.

Metal deactivators may be of the aryl thiazines, triazoles, or alkyl substituted dimercapto thiadiazoles (DMTD's), or mixtures thereof. Metal deactivators can be present in the amount of about 0.01 to 0.2 vol%, more preferably in the amount of about 0.02 to 0.15 vol%, most preferably in the amount of about 0.05 to 0.1 vol%.

Antiwear additives such as metal dithiophosphates (e.g., zinc dialkyl dithiophosphate, ZDDP), metal dithiocarbamates, metal xanthates or tricresylphosphates may be included. Antiwear additives can be present in the amount of about 0.05 to 1.5 vol%, more preferably in the amount of about 0.1 to 1.0 vol%, most preferably in the amount of about 0.2 to 0.5 vol%.

Pour point depressants such as poly(meth)acrylates, or alkyl-aromatic polymers may be included. Pour point depressants can be present in the amount of about 0.05 to 0.6 vol%, more preferably in the amount of about 0.1 to 0.4 vol%, most preferably in the amount of about 0.2 to 0.3 vol%.

Antifoamants such as silicone antifoaming agents can be present in the amount of about 0.001 to 0.2 vol%, more preferably in the amount of about 0.005 to 0.15 vol%, most preferably in the amount of about 0.01 to 0.1 vol%.

Lubricating oil additives are described generally in "Lubricants and Related Products" by Dieter Klamann, Verlag Chemie, Deerfield, Florida, 1984, and also in "Lubricant Additives" by C. V. Smalheer and R. Kennedy Smith, 1967, page 1-11, the disclosures of which are incorporated herein by reference.

EXPERIMENTAL

a) Lab Nitration Screener Test Results

A lab nitration screener test was used in initial testing to guide in the selection of antioxidants, and viscosity index improvers (VIIs). The test results identify a number of parameters for assessing the used oil performance, including viscosity increase, oxidation , nitration, TAN increase, and TBN

depletion. All measurements are reported so that large results (e.g., viscosity increase, or TBN depletion) represent greater levels of lubricant degradation. Thus, numerically lower results represent a measure of longer oil life. In each test, a Reference Oil is always tested. All results are reported as a ratio of the result for the Candidate Oil divided by the result for the Reference Oil. For example, if a Candidate Oil has an oxidation result of 1.0, then it has an oxidation performance equal to that of the Reference Oil. If the Candidate Oil has an oxidation result less than 1.0, then the Candidate Oil demonstrates oxidation performance superior to that of the Reference Oil.

The reference oil is an oil using as base oil a mixture of hydrocracked 500N base oil and solvent refined 1200N base oil, to which is added a pour point depressant and about 9.6 vol% of a commercial additive, Oloa 1255 (NGEO additive System B) which is believed to impart a net treat rate of about 0.4 vol% BHT (phenolic anti oxidant) in the formulated oil. Oloa 1255 is one of the most widely sold gas engine oil additive packages and represents, therefore, a "benchmark standard" against which other formulations may be measured.

EXAMPLES

Lab nitration screener test results are summarized in Table 1. A first series of results, candidate formulations 1 through 9, employ the identical core additive package (NGEO Additive System A), and are toptreated with different antioxidants. Results are measured relative to Reference Oil 1. The toptreats include antioxidants which are amine only, phenol only, and phenol/amine combinations (one of which is made up of about 80% aminic component, and 20% phenolic component, and the other of which is made up of about 70% aminic component and 30% phenolic component). Results show that the candidate oils with the longest life (i.e., lowest values of viscosity increase,

oxidation, nitration, TAN increase, TBN depletion) are those formulations including phenol only antioxidants. Indeed, the results with phenol only show the longest life, with all used oil parameters less than 1.0 (thus, all results show longer life relative to the Reference Oil).

A second series of results, Candidate Formulations 10 through 13, investigates the combined benefits of antioxidants and VIIs . It should be noted that the use of VIIs in multigrade gas engine oils has been established for over two decades. The present candidate formulations, however, differ in that they use low treats of VII as a thickener to simply boost the base formulation to a target viscosity of 13.2 cSt at 100°C. The low treat of VII allows a heavy 1200 SN solvent refined basestock (vis of about 20.8 cSt at 100°C) to be eliminated from the formulation, but is insufficient to have them considered as a multigrade oil. Candidate Formulation 10 differs from Reference Oil 1 only by using phenol only instead of amine only anti oxidant. Examining the lab screener results for Candidate Formulation 10 therefore confirms the benefits for using phenol antioxidant versus an amine antioxidant. Candidate Formulation 11 includes a monofunctional VII in addition to the components used in Candidate Formulation 10. Results show that the inclusion of the VII offers additional benefits for controlling viscosity increase, oxidation, and TAN increase.

It must also be pointed out that Candidate Oils 11 and 13 each also exhibited a considerable and unexpected improvement in the nitration level as compared against any other oil except Candidate Oil 10, which was similar in terms of nitration performance but substantially equivalent to the Reference Oil in TAN delta. By comparison, Candidate Oils 11 and 13 exhibited not only an improvement in oxidation but also an improvement in TAN delta relative to the Reference Oil, showing the overall superiority of the phenolic type anti oxidant-viscosity index improver combination of the present invention.

NGEO Additive System B is a gas engine oil additive package which is believed to contain BHT, a phenolic type anti oxidant, which gives a finished oil anti oxidant content of about 0.4 vol%. Thus, the presence of a phenol anti oxidant in gas engine oil is known. Further, multigrade oils containing solvent extracted base oil, Additive System B and a high treat rate of viscosity index improver (about 8-11 vol%) so as to meet a SAE J-300 specification, but also containing 70% amine/30% phenol type anti oxidant mix as a supplementary anti oxidant, is also known.

The formulations of the present invention distinguish over those earlier formulations in that they contain viscosity index improvers at a low treat rate, not in an amount sufficient to produce a multi grade product, and further, the exclusive anti oxidant used is of the phenolic type, any aminic anti oxidant being excluded from the formulation. That this last difference is significant is seen by comparing Candidate Oils 4, 5, 6 and 7 against Candidate Oils 8, 9 and by comparing Candidate Oils 11 and 13 (oils of the present invention) against Candidate Oil 12.

It is seen that the oils containing aminic anti oxidants generally exhibit a lesser degree of improvement in terms of viscosity increase, oxidation, nitration, TAN delta and TBN depletion as compared against oils containing only phenol (Oils 4-7 vs. Oils 8 and 9), and that when oils containing viscosity index improver are considered, such oil containing amine anti oxidants (Oil 12) is markedly inferior in all measurement categories as compared against Oils 11 and 13, formulations of the present invention.

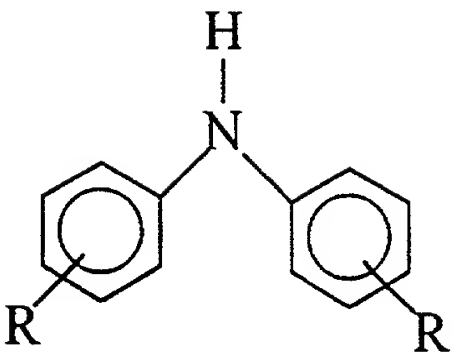
TABLE 1

CANDIDATE OIL		Ref Oil 1	1	2	3	4	5	6	7	8	9	10	11	12	13
NGEO additive system	(in vol%)		A	A	A	A	A	A	A	A	A	B	B	B	B
Additive System A *			← 6.98 →												
	pour point depressant	0.50	0.400	0.400	0.400	0.400	0.400	0.400	0.400	0.400	0.400	0.500	0.500	0.500	0.500
Additive System B		9.6	-	-	-	-	-	-	-	-	-	9.6	9.6	9.6	9.6
	Hydrocracked 500N base	77.55	87.120	87.120	87.120	87.120	86.620	87.120	86.620	87.120	86.620	82.200	88.150	88.380	88.200
	Solvent Refined 1200N base	11.35	5.000	5.000	5.000	5.000	5.000	5.000	5.000	5.000	5.000	6.700	-	-	-
Supplementary Antioxidants/Viscosity Index Improvers															
	Amine-1 only a/o	1.00	0.50	-	-	-	-	-	-	-	-	-	-	1.00	-
	Amine-2 only a/o	-	-	0.50	-	-	-	-	-	-	-	-	-	-	-
Irganox L118	Phenol-1 only a/o	-	-	-	0.50	-	-	-	-	-	-	-	-	-	-
	20% Phenol/80% Amine a/o Mix 1	-	-	-	-	0.50	1.00	-	-	-	-	-	-	-	-
	30% Phenol/70% Amine a/o Mix 2	-	-	-	-	-	-	0.50	1.00	-	-	-	-	-	-
Irganox L135	Phenol-2 only a/o	-	-	-	-	-	-	-	-	0.50	1.00	1.00	1.00	-	1.00
PTN 8024	Monofunctional OCP VII	-	-	-	-	-	-	-	-	-	-	-	0.75	-	-
TLA 6723	Multifunctional OCP VII	-	-	-	-	-	-	-	-	-	-	-	-	0.52	0.70

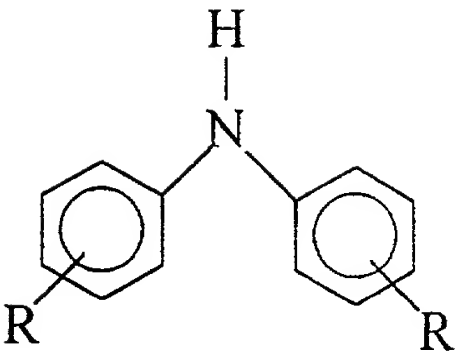
* Additive System A contains calcium phenate and calcium sulfonate detergents, a dispersant, a metal deactivator, an anti wear agent and an antifoamant, but no amine or phenol type anti oxidants.

TABLE 1 (continued)

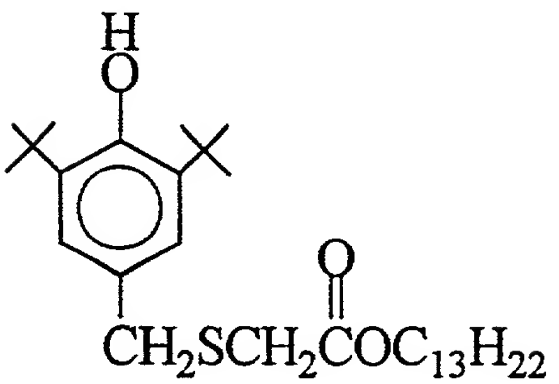
Results (Ratio:Candidate and/reference oil)															
Number of tests		-	1	1	1	1	1	1	1	1	1	2	2	2	2
% Visc Inc		1.00	0.56	0.52	0.39	0.55	0.63	0.57	0.72	0.36	0.44	0.91	0.78	0.81	0.76
Oxidation (abs/cm)		1.00	0.65	0.62	0.53	0.54	0.55	0.6	0.59	0.46	0.44	0.86	0.78	0.9	0.77
Nitration (abs/cm)		1.00	1.05	1.05	0.92	0.97	0.91	1.11	1.05	0.91	0.8	0.68	0.68	0.96	0.69
TAN Delta		1.00	1.57	1.41	2.14	0.66	0.51	1.22	2.28	0.54	0.47	1.02	0.76	1.34	0.75
TBN (%Depleted)		1.00	0.97	0.98	0.93	0.98	1.04	1.01	1.03	0.8	0.78	0.93	0.93	1.14	0.94
Oil Additive Packages		A =	Low Ash Additive system with 0.45% ash content												
		B =	Additive system with 0.45% ash content. It is believed that the												
			additive system imparts a net treat rate of about 0.4 vol% of BHT antioxidant in the formulated oil.												



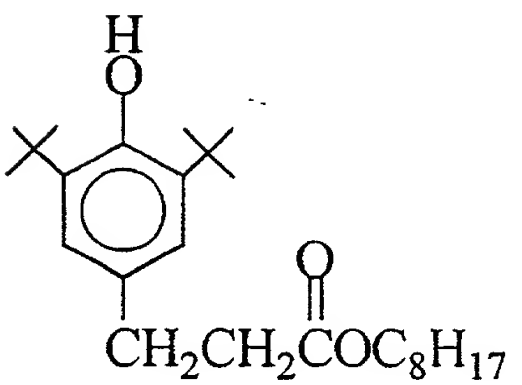
Amine 1
R = H, C_9 mix



Amine 2
R = C_4-C_8



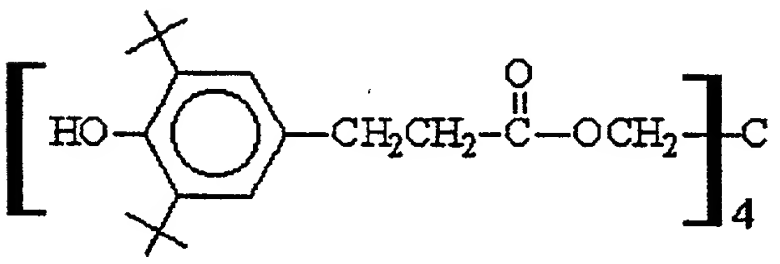
Phenol 1



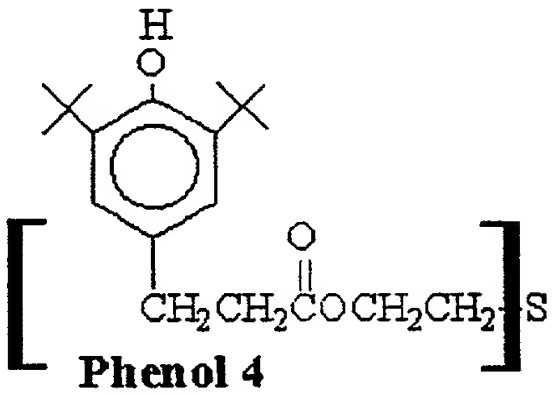
Phenol 2

Amine/Phenol Mix 1 is a mixture of Amine 2 and Phenol 4.

Amine/Phenol Mix 2 is a mixture of Amine 2 and a mixture of Phenol 4 and Phenol 3.



Phenol 3



Phenol 4

b) Lab Engine Test

Lab engine testing was completed to verify the screener results. The used oil properties from the engine test are summarized pictorially in

Figures 1A, 1B and 1C. The results are expressed in terms of absolute levels of viscosity increase (%) (Figure 1A), oxidation (absorbance units/cm) (Figure 1B), and nitration (absorbance units/cm) (Figure 1C). The results show duplicate test data for Reference Oil 1 (these are marked on the graphs as Reference Oil 1 - Test 1, and Reference Oil 1 - Test 2), and verify that the engine test provides repeatable results. Candidate Oil 11 is identified in Figures 1A, 1B and 1C as "Invention", and shows significant improvements in viscosity increase, oxidation and nitration relative to Reference Oil 1. For additional reference, two competitive oils (Commercial Oil 1 and Commercial Oil 2) are also shown on this graph. These two commercial oils are relevant, because they are strongly believed to be formulated with the same additive package (Additive System B), and also use severely hydrofinished basestocks.

c) Field Demonstration Results

Figure 2 shows results from a field demonstration of the invention. Reference Oil 2 uses Additive System B, and solvent extracted basestocks. Reference Oil 1 is identified in this graph as Reference Oil 1, and represents data collected over many years. The left data point for Reference Oil 1 (1190 hours) represents a drain interval prior to optimization of the engine tuning parameters (fuel mixture, timing, etc.). The right data point for Reference Oil 1 (1480 hours) represents the drain interval after optimization of the engine tuning parameters. More importantly, this second data point for Reference Oil 1 represents operation of the engine just prior to testing of the invention. The invention is identified in Figure 2 as "Invention", and identifies an oil drain interval of 2083 hours. These results demonstrate the benefits of the invention.

CLAIMS:

1. A long life gas engine oil comprising a major amount of an oil of lubricating viscosity and a minor amount of additives comprising phenolic anti oxidants and viscosity index improver, but which does not contain aminic anti oxidant, wherein the phenolic anti oxidant is present in an amount in the range of about 0.1 to 2 vol% and the viscosity index improver is present in an amount in the range of about 0.1 to 3 vol%.

2. The long life gas engine oil of claim 1 wherein the oil of lubricating viscosity is a natural oil, a synthetic oil or a mixture thereof having a viscosity of between about 5 to 20 cSt at 100°C.

3. The long life gas engine oil of claim 1 or 2 wherein the oil of lubricating viscosity has a viscosity of between about 7 to 16 cSt at 100°C.

4. The long life gas engine oil of claim 3 wherein the phenolic anti oxidant is present in an amount in the range of about 0.3 to 1.75 vol% and the viscosity index improver is present in an amount in the range of about 0.2 to 2 vol%.

5. The long life gas engine oil of claim 4 wherein the oil of lubricating viscosity has a viscosity of between about 9 to 13 cSt at 100°C, the phenolic anti oxidant is present in an amount in the range of about 0.5 to 1.5 vol% and the viscosity index improver is present in an amount in the range of about 0.3 to 1.5 vol%.

6. A method for enhancing the life of gas engine oils as evidenced by a reduction in viscosity increase, oxidation, nitration, TAN increase, and

TBN depletion, comprising adding to a gas engine oil comprising a major amount of an oil of lubricating viscosity but which does not contain aminic anti oxidant, a minor amount of a phenolic anti oxidant in the range of about 0.1 to 2 vol% and a minor amount of a viscosity index improver in the range of about 0.1 to 3 vol%.

7. The method of claim 6 wherein the oil of lubricating viscosity is a natural oil, a synthetic oil or a mixture thereof having a viscosity of between about 5 to 20 cSt at 100°C.

8. The method of claim 6 or 7 wherein the oil of lubricating viscosity has a viscosity of between about 7 to 16 cSt at 100°C.

9. The method of claim 8 wherein the phenol anti oxidant is added to the lubricating oil in an amount in the range of about 0.3 to 1.75 vol% and the viscosity index improver is added to the lubricating oil in an amount in the range of about 0.2 to 2 vol%.

10. The method of claim 9 wherein the oil of lubricating viscosity has a viscosity in the range of about 9 to 13 cSt at 100°C, the phenol anti oxidant is added in an amount in the range of about 0.5 to 1.5 vol%, and the viscosity index improver is added in an amount in the range of about 0.3 to 1.5 vol%.

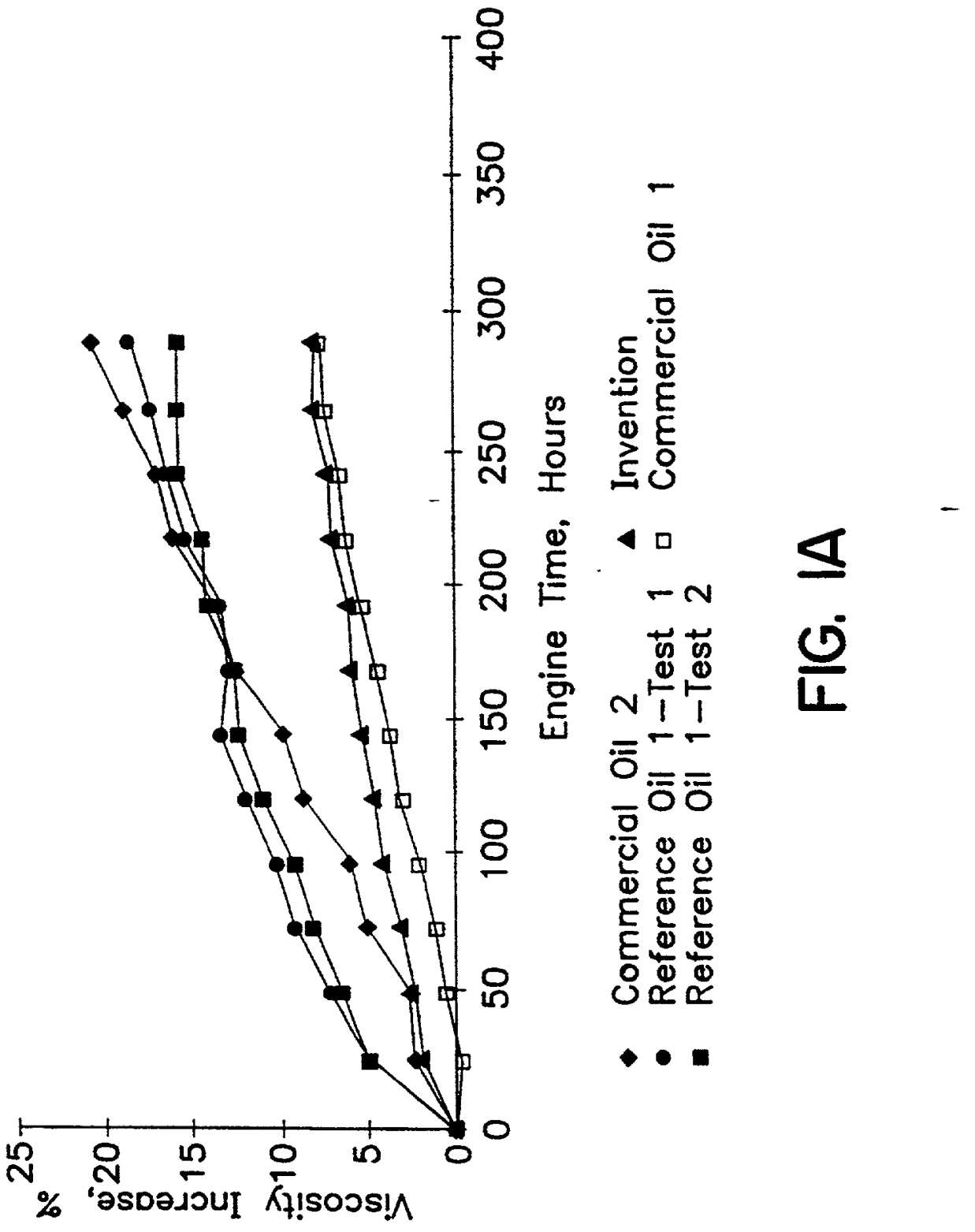

VISCOSITY INCREASE
Viscosity Increase, %
0
5
10
15
20
25
0
50
100
150
200
250
300
350
400
Engine Time, Hours
◆ Commercial Oil 2
● Reference Oil 1–Test 1
■ Reference Oil 1–Test 2
▲ Invention
□ Commercial Oil 1

FIG. 1A

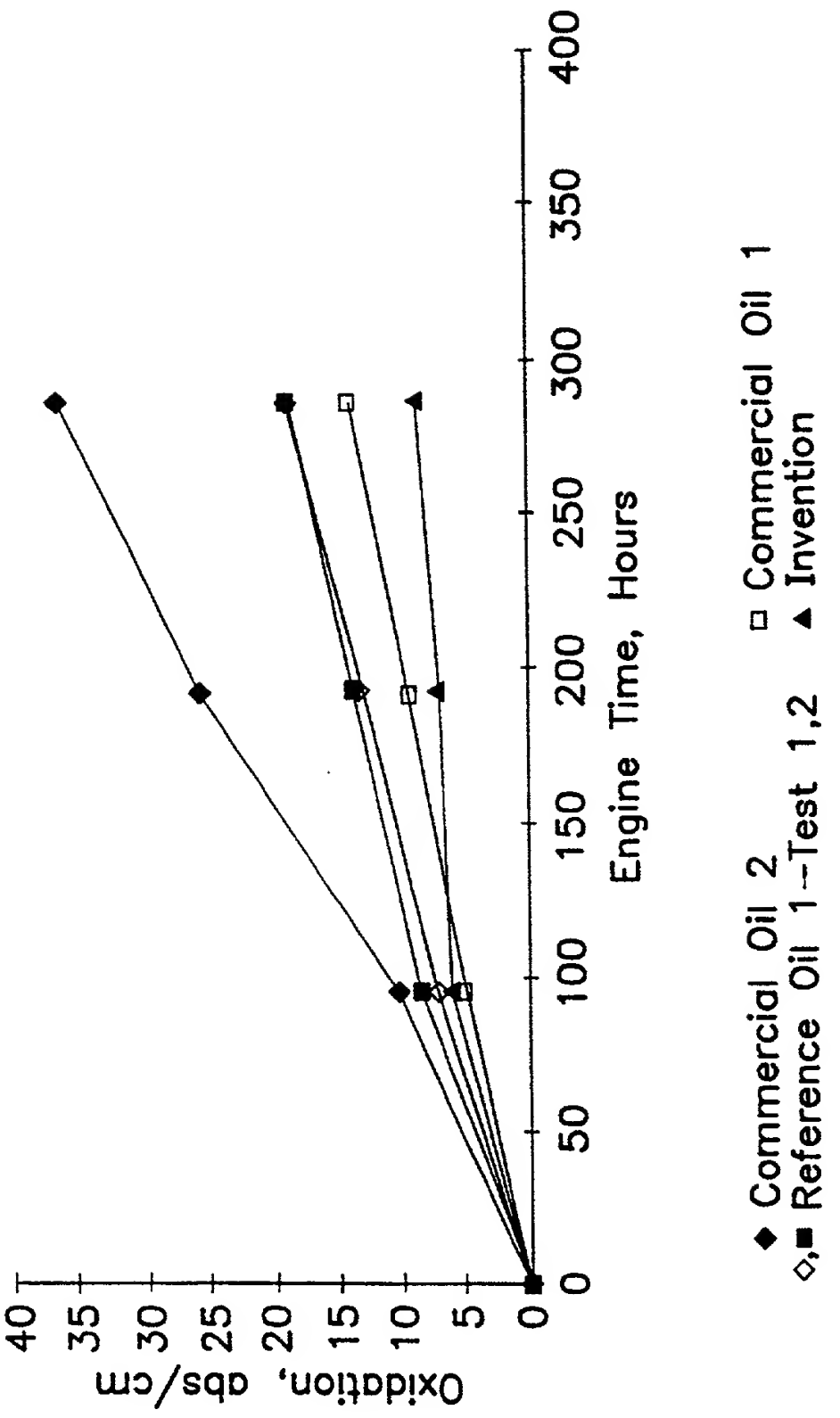

OXIDATION
Oxidation, abs/cm
0
5
10
15
20
25
30
35
40
0
50
100
150
200
250
300
350
400
Engine Time, Hours
◆ Commercial Oil 2
□ Commercial Oil 1
◇,■ Reference Oil 1–Test 1,2
▲ Invention

FIG. IB

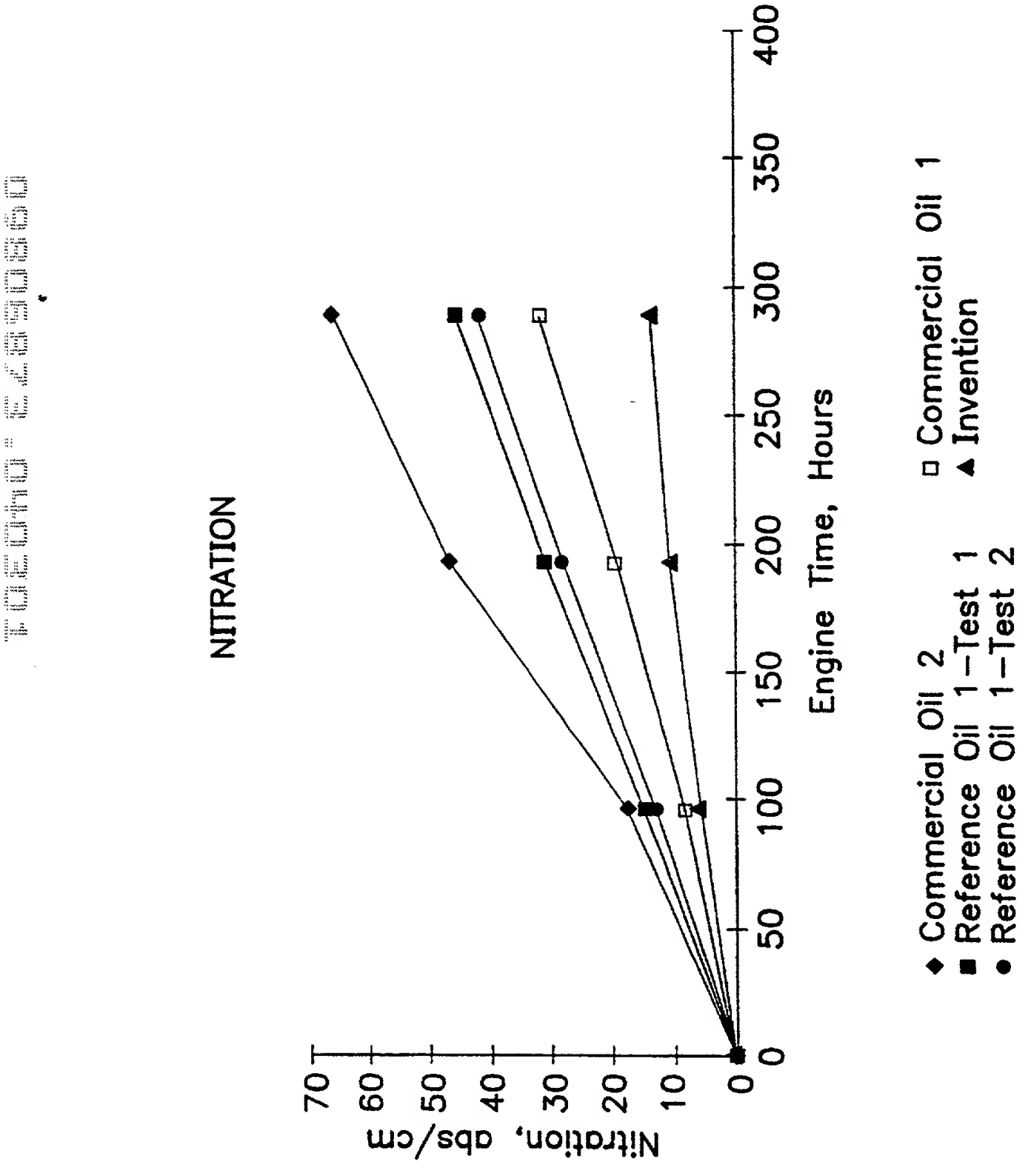

NITRATION
Nitration, abs/cm
0
10
20
30
40
50
60
70
0
50
100
150
200
250
300
350
400
Engine Time, Hours
◆ Commercial Oil 2
■ Reference Oil 1–Test 1
● Reference Oil 1–Test 2
□ Commercial Oil 1
▲ Invention

FIG. IC

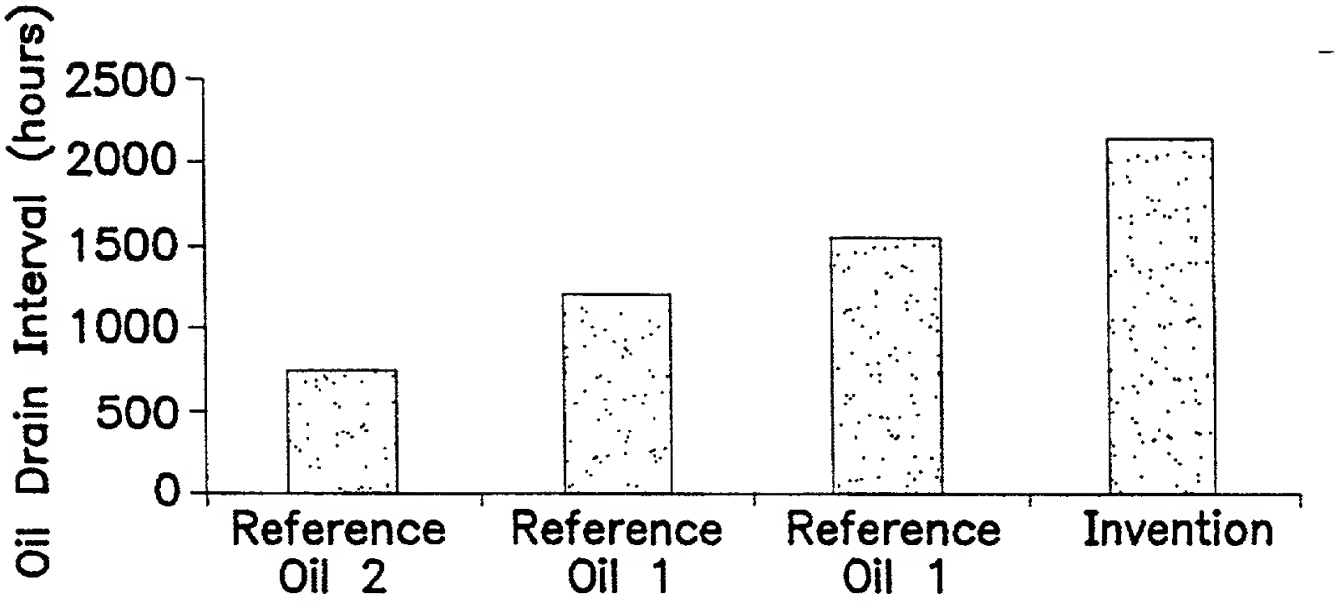

Oil Drain Interval (hours)
2500
2000
1500
1000
500
0
Reference Oil 2
Reference Oil 1
Reference Oil 1
Invention

FIG. 2

COMBINED DECLARATIO[illegible]R PATENT APPLICATION AND POWE[illegible]F ATTORNEY
(Includes Reference to PCT International Applications)

ATTORNEY'S DOCKET NUMBER
LAW771

As below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I an the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

"Long Life Gas Engine Oil and Additive System"

the specification of which (check only one item below):

☐ is attached hereto.

☐ was filed as United States application

Serial No.

on .

and was amended

on _____ (if applicable).

☒ was filed as PCT international application

Number PCT/US98/21595

on 13 October 1998.

and was amended under PCT Article 19

on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations. § 1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's cerrtificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. 119:

COUNTRY (if PCT indicate PCT)	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 USC 119	
			☐ YES	☐ NO
			☐ YES	☐ NO
			☐ YES	☐ NO
			☐ YES	☐ NO
			☐ YES	☐ NO
			☐ YES	☐ NO
			☐ YES	☐ NO

Combined Declaration For Patent Application and Power of Attorney (Continued)
(Includes Reference to PCT Internation Applications)

ATTORNEY'S DOCKET NUMBER: LAW771

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120:

U.S. APPLICATIONS		STATUS (Check One)		
U.S. APPLICATION NUMBER	U.S. FILING DATE	PATENTED	PENDING	ABANDONED

PCT APPLICATIONS DESIGNATING THE U.S.					
PCT APPLICATION NO.	PCT FILING DATE	U.S. SERIAL NUMBERS ASSIGNED (if any)			

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in teh Patent and Trademark Office connected therewith. (*List name and registration number*)

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2	FULL NAME OF INVENTOR	FAMILY NAME: CARTWRIGHT	FIRST GIVEN NAME: Stanley	SECOND GIVEN NAME: James
0	RESIDENCE & CITIZENSHIP	CITY: Sarnia	STATE OR FOREIGN COUNTRY: Canada CA	COUNTRY OF CITIZENSHIP: Canada
2	POST OFFICE ADDRESS	POST OFFCE ADDRESS: 2031 Huron Shores Court	CITY: Sarnia	STATE & ZIP CODE/COUNTRY: N7T 7H4
2	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
0	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
3	POST OFFICE ADDRESS	POST OFFCE ADDRESS	CITY	STATE & ZIP CODE/COUNTRY
2	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
0	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
4	POST OFFICE ADDRESS	POST OFFCE ADDRESS	CITY	STATE & ZIP CODE/COUNTRY

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

SIGNATURE OF INVENTOR 201	SIGNATURE OF INVENTOR 202	SIGNATURE OF INVENTOR 203	SIGNATURE OF INVENTOR 204
Alan Blahey			
DATE: MARCH 9, 1999	DATE: March 9, 1999	DATE	DATE

US

09/530773

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (Includes Reference to PCT International Applications)	ATTORNEY'S DOCKET NUMBER P1998J096

As below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I an the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

"LONG LIFE GAS ENGINE OIL AND ADDITIVE SYSTEM"

the specification of which (check only one item below):

☒ is attached hereto.

☐ was filed as United States application

Serial No.

on

and was amended

on _____ (if applicable).

☒ was filed as PCT international application

Number **PCT/US98/21595**

on **13 OCTOBER 1998**

And was amended under PCT Article 19

on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

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COUNTRY (if PCT indicate PCT)	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 USC 119	
			☐ YES	☐ NO
			☐ YES	☐ NO
			☐ YES	☐ NO
			☐ YES	☐ NO
			☐ YES	☐ NO

Combined Declaration For Patent Application and Power of Attorney (Continued)
(Includes Reference to PCT International Applications)

ATTORNEY'S DOCKET NUMBER: **P1998J096**

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120:

U.S. APPLICATIONS		STATUS (Check One)		
U.S. APPLICATION NUMBER	U.S. FILING DATE	PATENTED	PENDING	ABANDONED

PCT APPLICATIONS DESIGNATING THE U.S.					
PCT APPLICATION NO.	PCT FILING DATE	U.S. SERIAL NUMBERS ASSIGNED (if any)			

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (*List name and registration number*)

Joseph J. Allocca Registration No. 27,766
Paul E. Purwin Registration No. 29,203
Linda M. Scuorzo Registration No. 36,001

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201	RESIDENCE & CITIZENSHIP	CITY: Sarnia	STATE OR FOREIGN COUNTRY: Ontario, Canada	COUNTRY OF CITIZENSHIP: Canada
201	POST OFFICE ADDRESS	POST OFFICE ADDRESS: 1716 Trinity Crescent	CITY: Sarnia	STATE & ZIP CODE/COUNTRY: Ontario, Canada N7S 5K2
202	FULL NAME OF INVENTOR	FAMILY NAME: CARTWRIGHT	FIRST GIVEN NAME: Stanley	SECOND GIVEN NAME: James
202	RESIDENCE & CITIZENSHIP	CITY: Sarnia	STATE OR FOREIGN COUNTRY: Ontario, Canada	COUNTRY OF CITIZENSHIP: Canada
202	POST OFFICE ADDRESS	POST OFFICE ADDRESS: 2031 Huron Shores Court	CITY: Sarnia	STATE & ZIP CODE/COUNTRY: Ontario, Canada N7T 7H4
203	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
203	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
203	POST OFFICE ADDRESS	POST OFFICE ADDRESS	CITY	STATE & ZIP CODE/COUNTRY
204	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
204	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
204	POST OFFICE ADDRESS	POST OFFICE ADDRESS	CITY	STATE & ZIP CODE/COUNTRY

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SIGNATURE OF INVENTOR 201	SIGNATURE OF INVENTOR 202	SIGNATURE OF INVENTOR 203	SIGNATURE OF INVENTOR 204
Alan Blahey	Stanley Cartwright		
DATE: March 27, 2001	DATE: March 27, 2001	DATE	DATE

JJA:kak

9/26/00